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FABRICATION OF TUNGSTEN FOR SOLID-PROPELLANT
ROCKET NOZZLES

DEFENSE METALS INFORMATION CENTER
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FABRICATION OF TUNGSTEN FOR SOLID-PROPELLANT ROCKET NOZZLES

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SUMMARY

Tungsten as a material has been shown to have adequate properties to withstand the thermal, chemical, and mechanical environments in many of the more advanced solid-propellant rocket systems. Tungsten may be used in rocket components in a number of forms, but the leading form is pressed, sintered, and forged unalloyed tungsten. Most of the successful firings of full-scale nozzles at 5500 F or higher were accomplished with inserts of this type. Rapid progress is being made in the fabrication of sheet-tungsten liners backed up with graphite, and many future systems may employ this form of tungsten. An unexpected factor, which may lead to increased use of sheet tungsten, is the good performance of welded metal. Perhaps the most important problem connected with the use of sheet tungsten is that of bonding it to the backing material.

INTRODUCTION

One of the most serious problem areas in solid-propellant rocket motors is suitable materials for the throats of nozzles. This area is subjected to the maximum heat flux and maximum erosive attack in the motor system.

Of the refractory materials available, tungsten and graphite have proven to be the best for nozzles subject to the most severe environments. Of the two, tungsten has much better erosion resistance and is preferred for multiple nozzle motors where erosion rate is critical and where throats are moderately small.

The metal-fabrication industry has only recently acquired its first practical experience in preparing massive tungsten shapes and sheet in sizes large enough for rocket-nozzle construction. In the past couple of months work on the fabrication of tungsten, especially by forging melted as well as pressed and sintered material, has been accelerated rapidly. Many companies are currently engaged in this field. This memorandum does not attempt to detail all of these efforts. A DMIC report, dealing more extensively with this subject, is planned.

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The performance of nozzle inserts made from mill products has been strongly related to the specific fabrication processes used in consolidating the metal into massive form. It is the purpose of this memorandum to summarize information on (1) the consolidation and fabrication processes being used to produce large tungsten shapes and sheet, (2) the general physical characteristics of the various types of tungsten being shaped into rocket nozzles, and (3) the performance of these in solid-fuel rocket motors.

Molybdenum continues to have some applications, such as backup sleeves, where temperature environment is not so severe.

CONSOLIDATION

Tungsten generally is consolidated by powder-metallurgical processes. The classical self-resistance heating method developed by the lamp and electronic industry has been superseded by isostatic pressing in collapsible containers, followed by sintering in high-temperature furnaces or induction heating. Special shapes can be made by this procedure (Figure 1). There is a limitation to the section size which may be satisfactorily hydrostatically pressed due to the problem of obtaining reasonably uniform cold-pressed densities. However, sections 12 to 14 inches in diameter have been compacted successfully. At least for current billet sizes, compaction is not a limitation. Perhaps a more difficult problem is that of the indirect or furnace sintering of the pressed material. Up to recently, most of the sintering furnaces in the United States had a temperature limitation of about 2200 C. Austrian practice employed somewhat higher temperatures, believed to be at least 2500 C, and many people ascribed the reported superior performance of Austrian tungsten to this factor. However, most of the U. S. tungsten producers have, or are installing, furnaces with higher temperature capabilities. An as-sintered density of 90 per cent theoretical is generally considered to be the minimum acceptable for satisfactory workability in subsequent fabrication of the billet. There are serious furnace problems in achieving this density in large billets, for example the disproportionately long sintering time necessary at available lower temperatures. At present, the largest adequate indirect-sintered tungsten billet weighs about 140 pounds (Figure 2).

Hot pressing is further behind as a method of consolidating tungsten, primarily because of inadequate mold materials. Graphite is the only feasible material, and excessive carburization generally occurs during hot pressing in graphite. The resulting carburization makes it difficult to hot work or hot press into shape. However, some interesting work is being done currently on hot pressing tungsten directly to a graphite backing, and this may eliminate the necessity for workability. This process is called "integral pressing".

The arc-casting process was developed for molybdenum. Molybdenum ingots up to 16 inches in diameter, weighing 4,000 pounds, have been melted, extruded, and fabricated. Because of the large grain size of such ingots and



FIGURE 1. TUNGSTEN SHEET BAR SLAB MADE BY ISOSTATIC PRESSING
Sylvania.



FIGURE 2. TUNGSTEN FORGING BILLET 5 INCHES IN
DIAMETER AND 12 INCHES LONG

Sylvania.

the accompanying hot shortness, it has been a universal practice to extrude prior to other types of working. Extrusion breaks up and refines the as-cast structure. A major advantage of the arc-casting process is that there is no apparent limit other than electrical power on the size of the ingot that can be produced. For example, arc-melting furnaces up to 40 inches in diameter have been built for steel melting. Furnaces as large as 100 inches in diameter are feasible. Therefore, for refractory metals the arc-casting process offers the best possibilities if very large ingot sizes become needed.

Arc melting of tungsten has only recently been accomplished, and the arc casters have produced only relatively small ingots (Figure 3). Table 1 illustrates the ingot sizes achieved as of spring 1961 and Figure 3 illustrates a typical arc-cast tungsten ingot. Unquestionably, larger size ingots soon will be produced. As in the case of arc-cast molybdenum, a coarse structure results (Figure 4). Fast-acting extrusion presses are available with capacities up to 12,000 tons. These could be used to break down cast tungsten ingots in diameters up through about 12 inches. However, at present, heating furnaces with capacities comparable to their corresponding extrusion facilities are inadequate.

Tungsten castings for utilization as nozzles in the cast condition have chiefly been confined to tungsten-molybdenum alloys, particularly 85W-15Mo. Most of the material evaluated has been machined from as-cast vacuum-arc-melted ingots. Some effort has been devoted to centrifugal casting of this alloy to obtain finer grain size (Figure 5). Plans are extant to roll tungsten sheet directly from centrifugally cast metal. There is some interest in grain refining tungsten intended to be hot worked using about 2 per cent molybdenum or less than 1 per cent boron or other grain-refining additive.

Electron-beam melting is an even newer process, whose chief claim to fame is the extremely low interstitial content which results from melting in the ultrahigh vacuum required for the process and which should make possible mechanical working without a preliminary extrusion operation. The electron-beam process also results in extremely large grain size, larger even than in arc-cast ingots. Grain refining electron-beam melted tungsten to permit direct forging is in preliminary development, and it is not possible at this time to assess the future possibilities of this type of material. The development of electron-beam furnaces is proceeding quite rapidly, and megawatt furnaces capable of melting 20-inch steel, 16-inch columbium, or 12-inch tungsten ingots are being built. The largest EB tungsten ingot so far melted was 4 inches in diameter.

Consolidated tungsten for fabrication has been produced by the plasma spraying process. This material is relatively porous as sprayed and normally requires hydrogen sintering to achieve minimum density even for limited hot workability. The chief application so far has been to provide preforms which need not be symmetrical in shape. These are forged to about 30 per cent reduction to fully consolidate the sintered body. The plasma spraying process, which so far has been conducted in air, has been criticized on the basis that the impurity content (up to a few hundred ppm oxygen) has been excessive. Attempts are being made to conduct the process in an inert-atmosphere chamber. Also, there are good indications that the limited workability of plasma-sprayed tungsten can be overcome by using a gas-pressure-



**FIGURE 3. THREE-INCH-DIAMETER TUNGSTEN INGOT ARC MELTED
FROM A 1-INCH-DIAMETER TUNGSTEN ELECTRODE**

U. S. Bureau of Mines, Albany.

TABLE 1. FACILITIES AT WHICH TUNGSTEN AND/OR TUNGSTEN
ALLOY INGOTS IN DIAMETERS OF 4 INCHES OR LARGER
HAVE BEEN PREPARED BY COLD-MOLD CONSUMABLE-
ELECTRODE ARC MELTING

Facility	Maximum Ingot Diameter, -inches	
	Unalloyed Tungsten	Tungsten Alloy
Climax Molybdenum Company	9	12(85W-15Mo)
General Electric Company	4	-
Oregon Metallurgical Corporation	4	12(85W-15Mo)
Universal Cyclops Steel Corporation	4	12(85W-15Mo)
Wah Chang Corporation	4-1/2	4(85W-15Mo)
Westinghouse Electric Corporation	8	-
U. S. Bureau of Mines (Albany)	5	-



**FIGURE 4. TYPICAL COARSE INGOT STRUCTURE IN
ARC-CAST TUNGSTEN INGOTS**

U. S. Bureau of Mines, Albany.

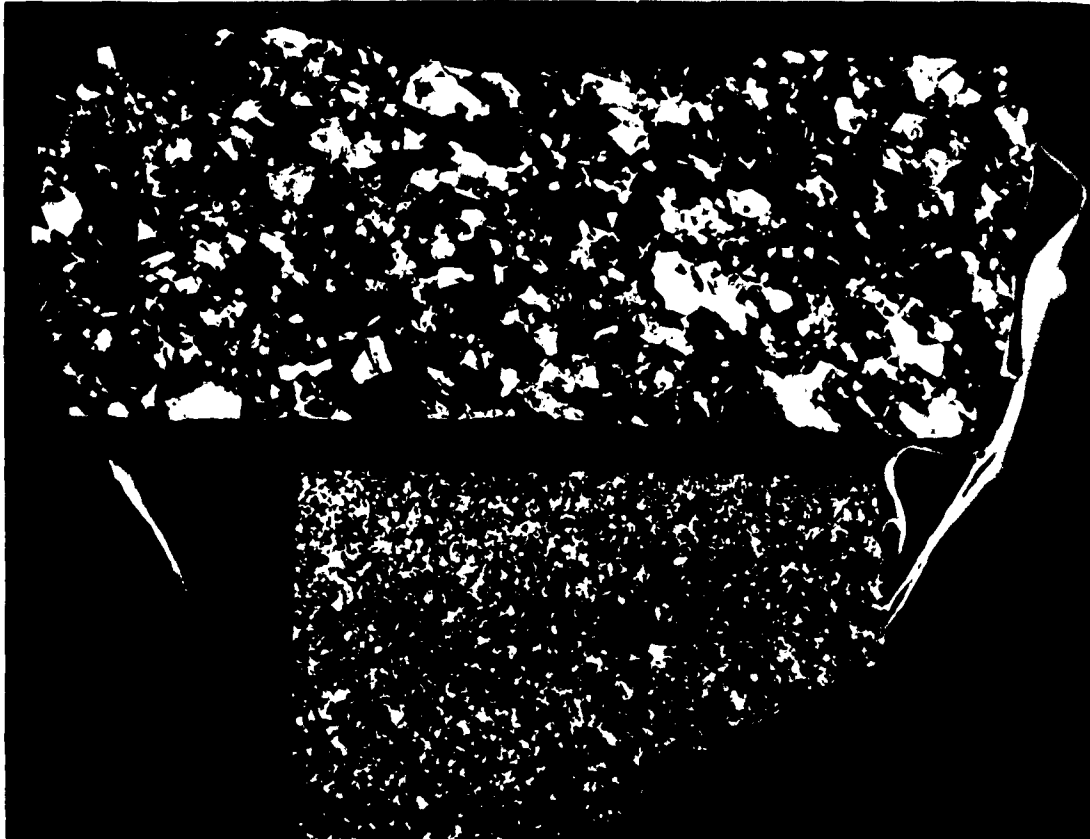


FIGURE 5. GRAIN SIZE OF 85W-15Mo ALLOY COLD-MOLD VACUUM ARC MELTED (ABOVE) AND SKULL MELTED AND CENTRIFUGALLY CAST (BELOW)

Oregon Metallurgical Corporation.

bonding process to densify the preforms prior to working. For economic reasons, however, it appears doubtful that plasma-sprayed tungsten as a consolidation process to produce ingots for subsequent mechanical working will ever be of great importance. Another more valuable potential of the process is for coatings which will be discussed later. Vapor deposition is also considered as a process for producing forging preforms, but little or no work has been done in this area. Except in special applications, preform production by this procedure is also not an economically competitive process.

PRIMARY FABRICATION

Powder-metallurgy-consolidated material generally is directly worked to the final desired shape. However, arc-cast or EB ingots require extrusion before they may be worked further.

Thus far, the extrusion of tungsten and its alloys has largely been confined to ingots of 3 inches or less because of the unavailability of furnaces for preheating larger billets to temperatures on the order of 3000 F. This has limited billet sizes for subsequent forging, and thus may limit the future possibilities of arc-cast tungsten in forged nozzle inserts. However, extruded arc-melted tungsten may well find its ultimate role in sheet bar to be processed into sheet.

Successes in the direct forging of tungsten nozzle inserts have largely been restricted to billets prepared by pressing and sintering. Apparently, closed-die forging is required in order to achieve the large reductions believed essential for good performance. Initially, some difficulties were encountered with inconsistencies in the forgeability of billets produced by various suppliers. This situation has improved with time although, at present, not all of the powder producers are able to produce sintered billets of 4-inch diameter or greater with consistently good workability. Difficulties were also encountered in developing closed-die forging procedures for tungsten. Currently, however, at least two forging companies (Ladish and Reisner) have developed proprietary techniques by which nozzle inserts and entrance caps up to 8 inches in diameter (Figure 6) are being successfully prepared.

So far, forging billets from arc-cast or electron-beam tungsten have not been produced in sizes large enough to be forged into nozzle inserts. Presumably, the lack of experience and/or equipment for preparing and extruding large-diameter tungsten ingots is the reason for this situation.

Recently, the first-known successes with the ring rolling of tungsten were reported by Reisner Forge Company. As illustrated in Figure 7, rings in diameters to 11 inches with 3/4-inch wall thickness have been rolled from pressed and sintered preforms. So far as is known, the actual ring rolling of nozzle shapes has not yet been accomplished although prospects for this appear very good.

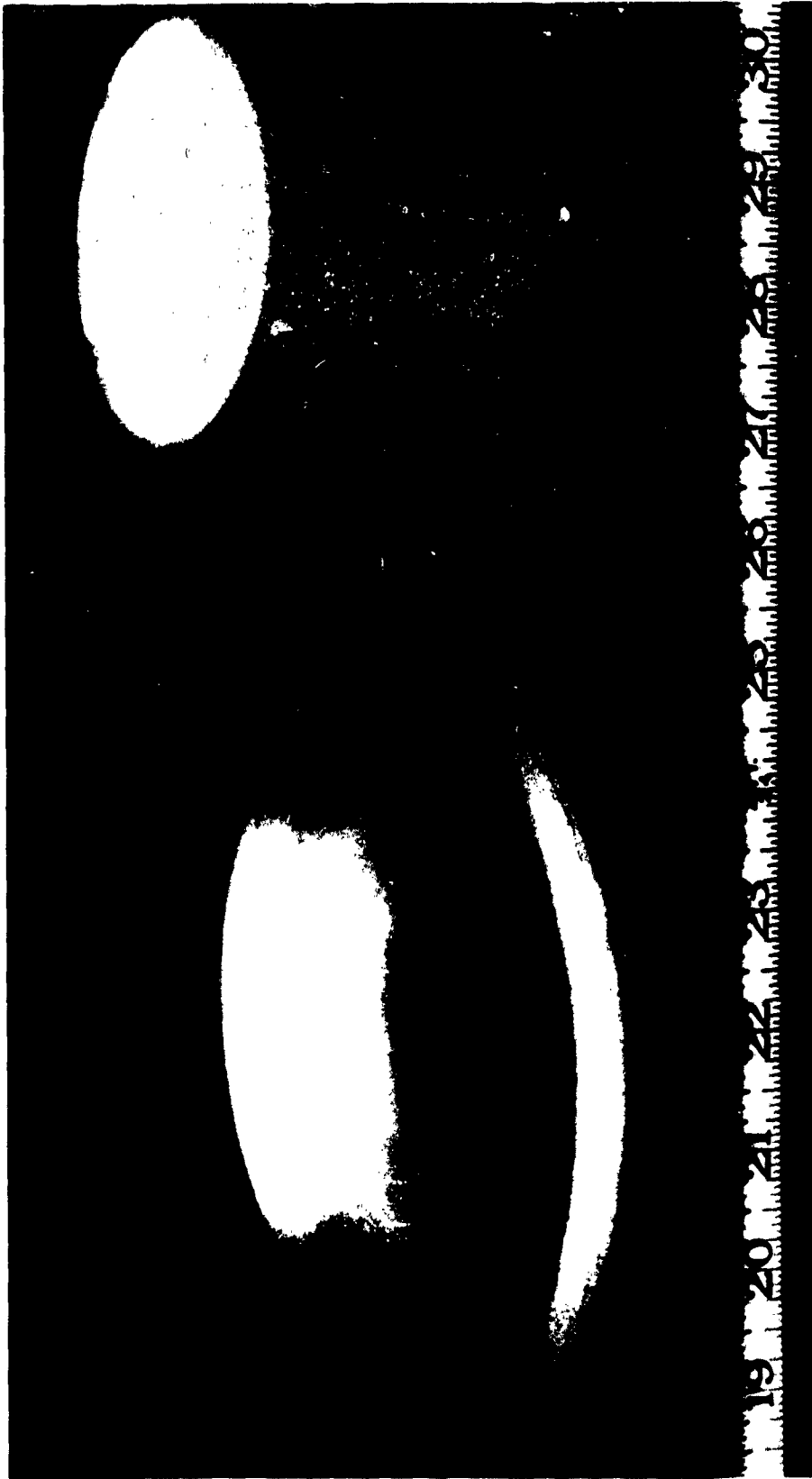


FIGURE 6. TUNGSTEN ENTRANCE CAP FORGED FROM PRESSED-AND-SINTERED BILLET

Reisner Forge Company.



FIGURE 7. TUNGSTEN RING ROLLED FROM PRESSED-AND-SINTERED PREFORM

Reisner Forge Company.

The subject of tungsten sheet has been confused with more conflicting statements on material origin and fabrication than perhaps any other subject in the nozzle field.

Fansteel is the leading domestic producer so far as sheet size is concerned. Up to recently, their maximum size sheet was 17 x 17 x 0.040 inch. However, at the Western Metals Congress in Los Angeles in March, 1961, Fansteel exhibited a tungsten sheet 18 to 20 inches wide by 3 feet long. G.E. has produced only small sheet sizes, but contemplates 36 x 96-inch sheets in their new facility being installed in Cleveland. Sylvania is said to be claiming 24-inch-wide tungsten sheets. There has been great interest in Austrian-produced sheet from Metallwerk Plansee. Their current production facilities are suitable for sheets up to 18 inches wide, but they are installing mill capacity which will permit 40-inch-wide sheets. They produced some pilot 40-inch-wide sheet in conjunction with Deutsche Edelstahlwerke in Krefeld.

The major limitations in producing wide sheets are small-size sheet bar and inadequate furnaces for sintering and preheating for rolling. Surprisingly, there has been little difficulty with mills capable of rolling wide tungsten sheet, probably because of the relatively low yield strength of the heated material. The 40-inch-wide sheet rolled in Germany was said to have been rolled on a two-high mill designed for stainless steel. The cold rolling of wide, thin sheet at room temperature may require robust mills.

Arc-cast tungsten may prove to be ideally suited for fabricating tungsten sheet. The ingot-size limitation by powder-metallurgy consolidation is not applicable to the cast product, and extruded arc-cast tungsten probably can be fabricated to a larger sheet bar than will be possible using the powder process if the necessary extrusion facilities are available. Also, the purity, particularly at grain boundaries, of arc-cast tungsten should be somewhat better than that of the powder-metallurgy material, if past experience with molybdenum holds true. Therefore, many of the inconsistencies that plague the fabricators of powder-metallurgy sheet tungsten at present may be overcome.

SECONDARY FABRICATION

Most of sheet-metal tungsten liners so far evaluated have been produced by conventional spinning, a process which does not reduce thickness. Therefore, large blanks are required in order to produce nozzles of large size. For conventionally spun long-type nozzles, it is estimated that the blank diameter has to be three times the throat diameter. Thus, for a 5-inch diameter nozzle, minimum 15-inch wide sheet is required. If tungsten nozzles with throat diameters significantly larger than those of the current generation are to be produced, much larger sheets will have to be rolled. About 36-inch-wide sheet is the largest contemplated for domestic production. This will permit nozzles of the long type with 12-inch-diameter throats. For short nozzles, a blank only about 1.3 times the throat diameter is required.

Relatively low temperatures (1800 to 2000 F) are being used for conventional spinning. These are not sufficient to recrystallize tungsten and correspond to warm working. Figure 8 illustrates a typical conventional spinning operation, wherein torch heat is applied to a tungsten-sheet blank as it is spun to the contour of a removable mandrel. Spinnability appears to be a function of sheet quality. Here is where there is confusion as to the sources of materials with adequate quality for good spinning characteristics. Hughes Tool has made a strong point that Austrian tungsten is much better than domestic as far as ability to spin is concerned. Marquardt, on the other hand, preferred domestic tungsten. In any case, neither domestic nor Austrian tungsten is considered adequate so far as reproducibility and consistency is concerned. Advances in sheet production technology are needed before the material may be said to be under control.

Shear spinning is less advanced than conventional and is a more costly process. During the shear-spinning operation, the forming tool not only forces the metal to conform to the shape of a rotating mandrel, but at the same time applies enough pressure to thin the sheet section. Large shear-spun nozzles (insets) are estimated to cost \$2500 each compared with \$1000 for those spun conventionally. The major advantage of shear spinning is that, because considerable reduction occurs in the operation, the size of the starting blank need not be so large. Usually the operation is done in stages over cores of increasing acuity. Some difficulty has been associated with annealing or stress relieving between stages, and it may be necessary to minimize thermal treatment. So far, all shear-spun tungsten evaluated by Aerojet-General Corporation has been domestic in origin. General Electric Company, doing development work on shear-spun large-diameter short nozzle inserts, found they could successfully shear spin only material of Austrian origin. It should be pointed out that, as of March, 1961, the shear spinning of long nozzle inserts from Austrian tungsten has not yet been attempted.

The temperatures employed in shear spinning are approximately the same as those for conventional spinning, and there appear to be no serious tooling or heating problems associated with the operation.

Explosive forming is in a more primitive stage, and is of interest mainly for asymmetrical nozzles. To provide sufficient temperature for explosive forming, liquid-metal baths (molten aluminum) are being employed as the hydraulic medium. So far as is known, no other high-energy forming methods are being contemplated for nozzle inserts.

Tungsten can be deep drawn if it is heated, and some work in fabricating full-scale nozzles by this process has been done. This includes the preparation of some complex nonsymmetrical parts, as illustrated in Figure 9, where welding has been used to assemble two or more separately formed components into a finished part.

Welding

The fusion welding of tungsten has been remarkably successful in view of the considerable difficulties encountered in welding molybdenum in the past. The major techniques are tungsten inert-gas-shielded (TIG) and



FIGURE 8. CONVENTIONAL SPINNING OF TUNGSTEN SHEET

Hughes Tool Company.

BATTELLE MEMORIAL INSTITUTE



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**FIGURE 9. FORMED FROM 0.060-INCH PURE TUNGSTEN SHEET
AND WELDED WITH PURE TUNGSTEN FILLER ROD**

Super-Temp Engineering and Manufacturing, Inc.

electron-beam welding (EB). Figure 10 illustrates a simulated rocket nozzle joined by electron-beam welding. Figure 11 summarizes strength comparison data for roll-formed tungsten and electron-beam welds in it. Note that, above the recrystallization temperature for the parent roll-formed tungsten sheet, welds are as strong or slightly stronger than adjacent material. This may be due in part to the purification of weld metal during EB melting. The processes are devised to minimize the grain size of the weldments. It had been noted in TIG welding that increasing the welding speed from 2 inches per minute to 16 inches per minute will result in grain sizes equivalent to EB weldments (corresponding to ASTM 3-4). Apparently, tungsten sheet does not have to be preheated prior to arc welding, and the weldments do not crack readily. The latter may, in part, result from the low coefficient of expansion. With relatively fine-grained weldments, it is possible to fabricate sheet-tungsten parts that have been fusion welded in much the same manner as the tungsten-base metal if discretion is used. This holds hope for greatly simplifying the problem of large tungsten sheets, since it may obviate the need for large-size tungsten-sheet blanks. Reportedly, welded tungsten can be bent or spun in much the same manner as the base metal. The bend transition temperature of wrought tungsten sheet is above 700 F, while that of the weldment is about 1100 F. If forming temperatures are maintained above the weld transition temperature, no allowance is made for the fact that the part being formed is welded. Welding would be particularly advantageous in conjunction with the fabrication of complex shapes and unsymmetrical parts which cannot be spun.

Brazing is primarily of interest in joining sheet-metal liners to graphite backing. Generally, the liner is joined by a graphitic-base cement. However, development work is being done on alloys of tungsten and molybdenum containing relatively large amounts of nickel and other low melting metals. These brazing materials are reported to have a much higher remelt temperature than the initial flow temperature. However, there is some question about the incipient melting temperature of such brazed parts. Full-scale firing tests will have to be performed before such joining practices can be approved. Titanium is also used to braze tungsten to graphite and forms a brazement which is substantially TiC. The advantage of titanium brazing is that the melting point of the brazed material is much higher than it is in the case of lower melting brazing metals. It is the usual practice in using brazed parts to anchor the sheet-metal insert mechanically as well as through the brazing material.

In many cases, it has been considered desirable to avoid the formation of carbide at the interface between tungsten and graphite, and rhenium has been suggested as a barrier material for this purpose. The technical basis for this suggestion is that rhenium does not form a carbide, although it dissolves appreciable carbon. The firing experience involving rhenium barriers between tungsten and graphite has been largely negative, and in some cases, rhenium barriers are believed detrimental because of formation of a low-melting alloy of tungsten, rhenium, and carbon.



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**FIGURE 10. SIMULATED TUNGSTEN ROCKET NOZZLE JOINED
BY ELECTRON-BEAM WELDING**

General Electric Company.

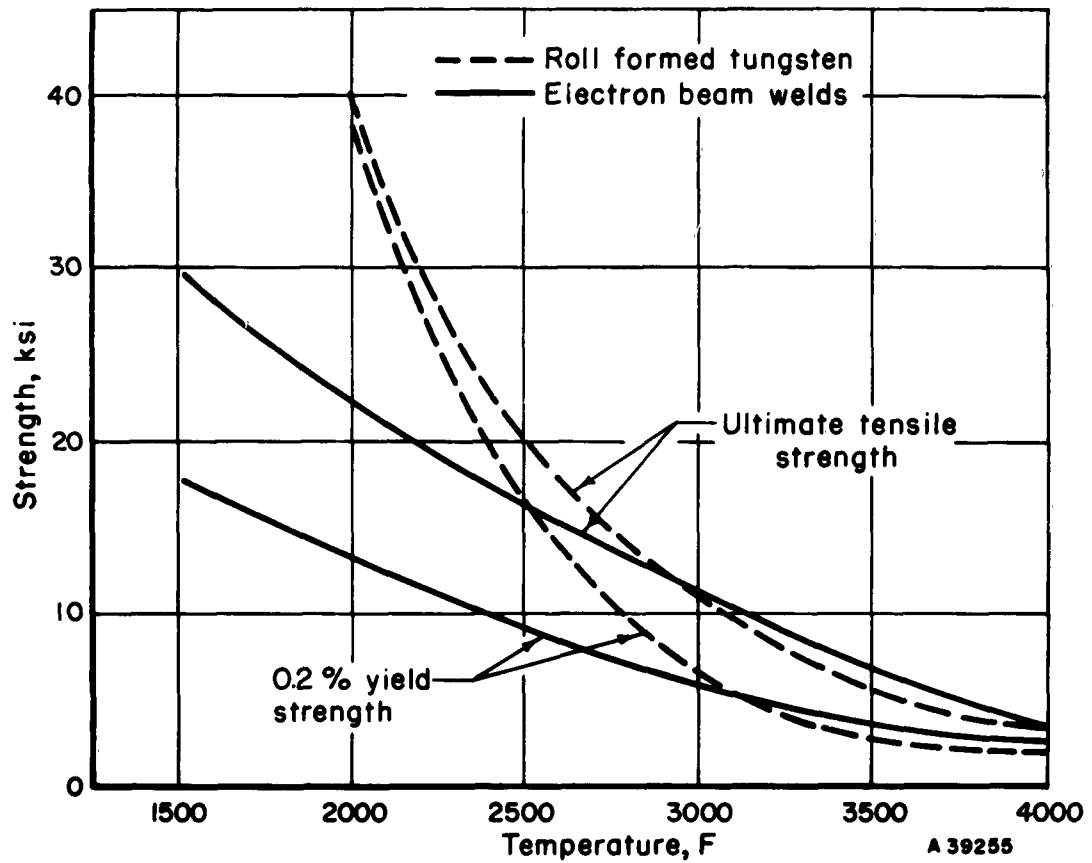


FIGURE 11. COMPARISON OF ELEVATED-TEMPERATURE TENSILE PROPERTIES OF ROLL-FORMED TUNGSTEN AND ELECTRON-BEAM WELDS IN TUNGSTEN SHEET

Machining of Tungsten

A discussion of fabrication of refractory metals for solid-rocket nozzle components would not be complete without mention of machining. In cast or forged nozzle inserts, the finished nozzle is only 10 to 20 per cent of the starting blank. For example, the forged tungsten nozzle insert for large-diameter nozzles weighs 85 pounds as forged and only 18 pounds after machining. This is no doubt the primary reason why large-diameter forged tungsten nozzle inserts may cost \$2300 compared to \$1000 for spun inserts, despite the base metal cost of \$7 to 8 per pound for forged stock and \$30 per pound for sheet.

Conventional machining of tungsten is a very difficult, time-consuming operation and efforts are being made to employ electrolytic or electrical-arc discharge processes. Work being done on chem milling of tungsten also appears to offer good possibilities. Electropolishing of tungsten sheet may improve low-temperature ductility and has been noted to reduce the bend transition temperature as much as 200 F. An advantage of slightly porous tungsten over dense forged tungsten is improved machinability. This is one reason why Aerojet-General Corporation considers 90 per cent density in forged powder-metallurgy nozzle inserts to be optimum.

Deposition

Plasma or vapor-deposition processes have not yet made great progress, so far as production applications are concerned. However, considering the difficulties of depositing dense workable tungsten, the state of the art has been greatly advanced within the last year. As noted earlier, recent developments in plasma spraying are directed toward improving purity of the product through inert chamber spraying and to improving density (and hopefully workability) through gas pressure bonding. These may well bring the plasma process to a place where it will be quite useful, although it is questionable whether this process can compete economically with other methods of preparing symmetrical tungsten shapes. For unsymmetrical shapes it may be advantageous.

Vapor deposition is somewhat further behind as a method of depositing tungsten on a substrate, usually graphite. Proponents of the process claim the economics of deposition compare favorably with those for powder metallurgy as a method of producing tungsten shapes. The real advantage of both plasma and vapor-deposition processes is the ability to deposit on nonsymmetrical shapes. They are of considerable interest and importance in such nozzle developments.

PERFORMANCE

Massive Tungsten

Based on full-scale firing tests at high pressures, the most reliable material available for nozzle inserts is pressed, sintered, and forged tungsten. In nozzles for systems employing somewhat smaller nozzles fired at low pressures, where the environment is considerably less severe, it is possible to use pressed and sintered tungsten with above minimum (80 per cent) density. In a pressed, sintered, and forged tungsten nozzle of moderate size, some porosity is actually considered desirable as a means of improving machinability and thermal-shock resistance (through lowered modulus of elasticity). However, for the larger nozzle inserts operating at high pressure, the only successful material seems to be a high-density forged tungsten. The mode of failure for sintered and forged tungsten nozzle inserts is normally through thermal shock. Apparently, the surface heats up very rapidly and places a tensile stress on the back side which may initiate circumferential cracks oriented at a 45-degree angle to the exterior surface. The erosion and heat resistance of tungsten is so good that there is little damage on the interior surface subject to flame impingement.

Large cast tungsten nozzles made of 85W-15Mo have been successfully fired in high-pressure systems. Indeed, 85W-15Mo presently is the only nozzle insert material to have passed reliability and the full-scale flight test. However, the cast tungsten nozzle has lost favor because of excessive brittleness and susceptibility to thermal cracks on the back side of the nozzle insert. In order to provide sufficient strength for handling, the cast 85W-15Mo nozzle insert must be maintained at a minimum section size of about 0.5 inch, which makes it much too heavy. Despite its mass, it has been noted to deform under firing conditions. The mode of failure in 85-15 nozzles includes grain-boundary separation as well as thermal cracking. Attempts to reduce the grain size of 85-15 through centrifugal casting have not resulted in significant reduction in the minimum section needed for handling. Pure tungsten may be cast to shape but has such large grain size that it offers even greater handling difficulty than the W-Mo alloy.

Plasma-sprayed tungsten nozzle inserts have been produced by forging plasma-sprayed and hydrogen-sintered preforms. Generally the reduction in forging is relatively low, about 30 per cent, only little more than is required to reduce the porosity, and apparently is not sufficient to improve the performance under severe conditions. The main nozzle application in which plasma-sprayed tungsten nozzles have had serious evaluation and have been satisfactory is at low chamber pressure, but not at full (high) pressure.

Some subscale vapor-deposited nozzles have been successfully fired, but no full-scale nozzles of this type have been tested. It appears that the mechanical properties of tungsten nozzles prepared by current plasma-spraying or vapor-deposition techniques are inadequate to permit their application in large nozzles where thermal stresses are significant.

Sheet Tungsten

Sheet tungsten is of major importance for nozzle inserts, jetavators, blast tubes, and entrance caps (Figure 12). The most demanding application is nozzle inserts. The smaller-diameter inserts have been successfully fired, and it is probable that there will be applications of sheet-metal inserts of throat diameters of about 5 inches. These inserts are joined to graphite backing by graphitic or other types of cementing devices and have been successful in both low- and high-pressure full-scale firing with flame temperatures up to 6200 F. The main performance requirement seems to depend on the quality of the joint between the nozzle insert and the graphite backing. In most cases, it is necessary to seat the graphite sheet insert to its backing, similar to the seating of valves. There is some thought that the producers of sheet-metal liners will also provide the first layer of graphite backing cemented to the liner itself.

Larger sheet-metal liners may offer a problem since the thermal expansion that occurs during firing may result in a longitudinal buckling along the axis as a result of the inability of the graphite backing to expand at the same rate as the tungsten-sheet insert. It is hoped to overcome this problem through cushions which will absorb the expansion of the insert. The use of graphite cloth bonded with phenolic resins is one step in this direction. Also, the use of interposed layers with intermediate expansion characteristics offers some promise. Another alternative is to increase the section size of the sheet-metal insert so that it has sufficient compressive strength to avoid buckling. Also, it may be possible to use a ribbed, stiffened structure to reduce buckling.

Sheet tungsten may be used to line blast tubes to reduce erosion of graphite. Entrance caps of spun or drawn tungsten sheet have been successfully used. It is possible that some of the exit cone area may be lined with tungsten sheet as a means of reducing the erosion of graphite.

Jetavator parts have been made of either arc-cast or sintered molybdenum forged to size. A tungsten heat shield is used to resist the direct flame impingement. Originally, the Ta-10W alloy was used to back up the graphite, but this was changed to forged molybdenum because of cost. At the present time, forged molybdenum is used for backup sleeves in some nozzles.

COMPOSITES CONTAINING TUNGSTEN

A host of materials have been evaluated as nozzle-component materials for solid-rocket motors. Many of these have been discarded because of inadequate melting point or thermal-shock resistance. A number of materials remain in development stages and are being seriously considered for advanced systems. These are largely modifications of current successful nozzle materials. Two general types of composites containing tungsten have shown a measure of success and are being further developed. One type is tungsten based, to which other materials are added to improve properties such as the thermal-



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FIGURE 12. PURE TUNGSTEN ENTRANCE CAP FORMED TO PRECISION CONTOUR FROM 0.100-INCH SHEET

Super-Temp Engineering and Manufacturing, Inc.

shock heat-absorbing capacity of composites. The other type is refractories to which tungsten is added to supply structural integrity, particularly resistance to thermal shock.

Tungsten melts at 6200 F and is successfully used with 6200 F flame temperatures. The surface temperature of the nozzle approaches the flame temperature, and it is apparent that something will have to be done to modify tungsten if it is to be used successfully with flame temperatures higher than 6200 F without changing nozzle design. The major modifications on which development work is being done are to add insoluble materials which will absorb heat during firing and thereby reduce the surface temperature. One proposed method which has received fairly advanced evaluation is the addition of about 5 volume per cent BeO to tungsten. The idea behind the BeO addition is that, at high temperatures, tungsten and BeO react, forming tungsten oxide and beryllium vapor with the absorption of heat. Firing tests of W-BeO in large first-stage nozzle inserts did not show advantage over the very successful sintered and forged nozzle insert, and it is not likely that this material will be brought along further, at least for the current generation of nozzles. Addition of Al_2O_3 instead of BeO to tungsten also is of interest. Additions of nickel, copper, silver, iron, and other relatively insoluble alloying elements which form a second phase which wets the tungsten grain boundaries are being evaluated as sacrificial ablative additions which will flow to the surface and evaporate during firing, thus lowering the temperature. Some of these materials are produced by powder-metallurgical processes and are very useful in sintering large sections because of the accelerated rate of sintering that occurs in the presence of liquid phases. So far, such nozzle-insert materials have not been evaluated by firing tests. Another class of ablative additions includes lithium, lithium hydride, and magnesium hydride, infiltrated into porous tungsten.

Tungsten is also being modified by molybdenum wire reinforcing. The purpose of this modification is to lower the density and modulus of elasticity. Although it looks promising in early development, it is doubtful whether it would be applied in the current generation of nozzles.

CARBIDE-BASE COMPOSITES

Carbides are our most refractory solid materials; that is, they have the highest melting points. Carbides such as TaC, HfC, $4Ta \cdot 1ZrC$, and $4Ta \cdot 1HfC$ have melting points above 7000 F. Hopes of using these ultra-refractory materials in nozzle-component applications have been dimmed because of their poor thermal-shock resistance, a characteristic thought to be largely the result of their high moduli of elasticity and excessive brittleness. Attempts, therefore, are being made to modify the carbides to improve thermal shock. These have pretty much been resolved in favor of incorporation of tungsten, both foil and wire, in the carbide. One form is to use a tungsten egg crate to hold the carbide sectors. This has the advantage of reducing the section size of the carbide and correspondingly the acting thermal stress. So far, these developments are in early stages and no performance data are available on full-scale nozzles.

FUTURE TRENDS IN NOZZLE MATERIALS

Up to the present, we have been considering hot nozzle materials, where the melting point is the same or higher than that of the flame temperature, thus permitting mechanical stability even under conditions of thermal equilibrium. A forecast relating environment to materials, developed by Aerojet-General Corporation, follows:

- (1) Up to 1961, flame temperatures will be 5500 to 6200 F and nozzles may be designed on heat-sink principles, with the surface temperature of the nozzle below the melting point of the exposed material.
- (2) From 1961 to 1965, flame temperatures will rise from 6200 F to 7000 F, and nozzles will have to incorporate features such as sacrificial (ablative) additions.
- (3) Beyond 1965, flame temperatures will exceed 7000 F and it will be necessary to use cooled nozzle structures.

It is considered by some materials-oriented people that the alternatives to the heat-sink principle are so difficult that it may be desirable to fix flame temperatures to 6200 F maximum, continue with tungsten nozzles, and adjust other conditions to gain additional thrust. It must always be kept in mind that increases of flame temperature may result in increases in weight of nozzle structure, particularly if they have to be cooled. A point may ultimately be reached where the added weight is more detrimental than the improvements in specific impulse associated with higher flame temperatures.

Effects of Future Systems on Materials

There appear to be three main future systems changes which will have important effects on nozzle materials. The first two are changes in vector controls such as asymmetrical nozzles and fluid injection. The third is the development of large nozzles.

Guidance through vector controls, such as jetavators, or gimbaling devices, entail considerable operational difficulty. There is a strong desire, particularly on the part of the designers, to simplify control systems for solid rocket systems. The most advanced of these devices is the asymmetric, rotatable nozzle. Guidance in this case is obtained through simple rotation. While the asymmetric nozzle delights the designer, it horrifies the materials man. Under these conditions, the presently successful sintered and forged tungsten nozzle, or the symmetrical spun-tungsten-sheet nozzle, may have to be set aside and methods of fabricating asymmetric nozzles developed. The possibilities here include plasma spraying and vapor deposition, which are considered inferior from a materials standpoint to sintered

and to forged and spun. Another unpalatable alternative is to contour form the half parts and weld them together. Another method proposed for producing asymmetric nozzles from sheet is through explosive forming. These then would have to be very carefully backed and cemented to graphite. There appears little doubt but that the asymmetric nozzle will cause considerable increase in the cost of producing satisfactory nozzles. All of the methods currently used for symmetric nozzles no doubt could be adapted to asymmetric nozzles, but at considerable difficulty and great scrap rate and great cost.

A change in nozzle design that will be welcomed by the materials people is the use of gas injection for vector control, which would eliminate jetavators, gimbals, or asymmetric nozzles. This is being considered. In this case, the nozzle is symmetric, no joints are required, and there are no projections into the gas jet. Another alternative being considered is the use of Cannard motors on the upper stage to provide the vector control needed.

Large-Diameter Nozzles

There are possibilities that nozzles with much larger diameters will be required in the future. This could result from a change of the current concept of using clusters of small nozzles to one of using a single large nozzle. Also, there is need for very large boosters, particularly in space-vehicle systems. In any case, it may be necessary to develop a new generation of nozzles with diameters significantly larger than the current 4 to 9-inch throat diameter. In fact, preliminary studies are being made of the feasibility of large thrust motors of throat diameters up to 100 inches. The materials problems associated with the single large-diameter nozzles actually may be less severe than with the current generation because the detrimental effects of erosion which currently prevent graphite from being used in the throat would be largely eliminated. Erosion of 1 inch from the graphite surface which would enlarge the throat diameter of a 9 to 10-inch nozzle by 20 per cent would only enlarge a 100-inch nozzle by 2 per cent.

There is no doubt that refractory metals will be used in large-diameter nozzles, although not probably in the largest. If this is the case, the refractory metals will be required in sheet form, since it is unfeasible to consider forging blanks much in excess of 16 inches. If forged tungsten is to be used in such systems, it would be necessary to employ segmentations which would have an advantage in that the thermal stresses would be considerably reduced. It is probable that currently contemplated tungsten-sheet capabilities will be satisfactory for producing sheet-metal liners no matter what the diameter because of the success of tungsten welding.

Propellant Changes

If propellant temperatures go significantly higher, as is considered likely, ablative methods of keeping the tungsten-metal surface cool as previously discussed will have to be developed. If these developments

are successful, there should be little change in the type of materials used in nozzles for flame temperatures up to 7000 F. It appears likely that increases of flame temperature above 7000 F will be obtained with the use of fluorine, in which case 8000 to 8300 F temperatures may be encountered initially. Under these conditions, it will be necessary to employ cooled nozzle structures, most likely with liquid metals such as lithium or copper. Great changes in nozzle design and materials can be expected if this comes about. It is possible that sheet tungsten will continue to be used because of its good welding characteristics. However, it may be desirable to use columbium and tantalum, which are much easier to weld and more amenable to the production of complex welded assemblies of the type that would be required in a liquid-metal-cooled nozzle structure. The considerable backlog of information on handling liquid metals will be most valuable in this connection. Tungsten, tantalum, and molybdenum have somewhat better corrosion resistance to liquid metals than does columbium.

There are possible metal additives, like beryllium, to replace aluminum in the ammonium perchlorate-type propellants. Beryllium adds to specific impulse but also adds increased cost and toxicity. If beryllium is used, indications are that it will not entail any more severe corrosion environment than is being encountered with the aluminum additive.

If the propellant should be changed such that the exhaust were oxidizing, this would present perhaps insuperable materials problems, and might set the nozzle materials picture back to scratch.

LIST OF DMIC MEMORANDA ISSUED
DEFENSE METALS INFORMATION CENTER
Battelle Memorial Institute
Columbus 1, Ohio

Copies of the technical memoranda listed below may be obtained from DMIC at no cost by Government agencies and by Government contractors, subcontractors, and their suppliers. Others may obtain copies from the Office of Technical Services, Department of Commerce, Washington 25, D. C.

A list of DMIC Memoranda 1-90 may be obtained from DMIC, or see previously issued memoranda.

DMIC Memorandum Number	Title
91	The Emittance of Titanium and Titanium Alloys, March 17, 1961, (PB 161241 \$0.50)
92	Stress-Rupture Strengths of Selected Alloys, March 23, 1961, (AD 255075 \$0.50)
93	A Review of Recent Developments in Titanium and Titanium Alloy Technology, March 27, 1961, (PB 161243 \$0.50)
94	Review of Recent Developments in the Evaluation of Special Metal Properties, March 28, 1961, (PB 161244 \$0.50)
95	Strengthening Mechanisms in Nickel-Base High-Temperature Alloys, April 4, 1961, (PB 161245 \$0.50)
96	Review of Recent Developments in the Technology of Molybdenum and Molybdenum-Base Alloys, April 7, 1961, (PB 161246 \$0.50)
97	Review of Recent Developments in the Technology of Columbium and Tantalum, April 10, 1961, (PB 161247 \$0.50)
98	Electropolishing and Chemical Polishing of High-Strength, High-Temperature Metals and Alloys, April 12, 1961, (PB 161248 \$0.50)
99	Review of Recent Developments in the Technology of High-Strength Stainless Steels, April 14, 1961, (PB 161249 \$0.50)
100	Review of Current Developments in the Metallurgy of High-Strength Steels, April 20, 1961, (PB 161250 \$0.50)
101	Statistical Analysis of Tensile Properties of Heat-Treated Mo-0.5Ti Sheet, April 24, 1961, (AD 255456 \$0.50)
102	Review of Recent Developments on Oxidation-Resistant Coatings for Refractory Metals, April 26, 1961, (AD 255278 \$0.50)
103	The Emittance of Coated Materials Suitable for Elevated-Temperature Use, May 4, 1961, (AD 256479 \$2.75)
104	Review of Recent Developments in the Technology of Nickel-Base and Cobalt-Base Alloys, May 5, 1961, (AD 255659 \$0.50)
105	Review of Recent Developments in the Metallurgy of Beryllium, May 10, 1961, (AD 256206 \$0.50)
106	Survey of Materials for High-Temperature Bearing and Sliding Applications, May 12, 1961, (AD 257408 \$2.00)
107	A Comparison of the Brittle Behavior of Metallic and Nonmetallic Materials, May 16, 1961, (AD 258042 \$0.50)
108	Review of Recent Developments in the Technology of Tungsten, May 18, 1961, (AD 256633 \$0.50)
109	Review of Recent Developments in Metals Joining, May 25, 1961, (AD 256852 \$0.50)
110	Glass Fiber for Solid-Propellant Rocket-Motor Cases, June 6, 1961
111	The Emittance of Stainless Steels, June 12, 1961
112	Review of Recent Developments in the Evaluation of Special Metal Properties, June 27, 1961
113	A Review of Recent Developments in Titanium and Titanium Alloy Technology, July 3, 1961

LIST OF DMIC MEMORANDA ISSUED
(Continued)

DMIC Memorandum Number	Title
114	Review of Recent Developments in the Technology of Molybdenum and Molybdenum-Base Alloys, July 5, 1961
115	Review of Recent Developments in the Technology of Columbium and Tantalum, July 7, 1961
116	General Recommendations on Design Features for Titanium and Zirconium Production-Melting Furnaces, July 19, 1961
117	Review of Recent Developments in the Technology of High-Strength Stainless Steels, July 14, 1961
118	Review of Recent Developments in the Metallurgy of High-Strength Steels, July 21, 1961
119	The Emittance of Iron, Nickel, Cobalt and Their Alloys, July 25, 1961
120	Review of Recent Developments on Oxidation-Resistant Coatings for Refractory Metals, July 31, 1961
121	Fabricating and Machining Practices for the All-Beta Titanium Alloy, August 3, 1961
122	Review of Recent Developments in the Technology of Nickel-Base and Cobalt-Base Alloys, August 4, 1961
123	Review of Recent Developments in the Technology of Beryllium, August 18, 1961
124	Investigation of Delayed-Cracking Phenomenon in Hydrogenated Unalloyed Titanium, August 30, 1961
125	Review of Recent Developments in Metals Joining, September 1, 1961
126	A Review of Recent Developments in Titanium and Titanium Alloy Technology, September 15, 1961
127	Review of Recent Developments in the Technology of Tungsten, September 22, 1961
128	Review of Recent Developments in the Evaluation of Special Metal Properties, September 27, 1961
129	Review of Recent Developments in the Technology of Molybdenum and Molybdenum-Base Alloys, October 6, 1961
130	Review of Recent Developments in the Technology of Columbium and Tantalum, October 10, 1961
131	Review of Recent Developments in the Technology of High-Strength Stainless Steels, October 13, 1961
132	Review of Recent Developments in the Metallurgy of High-Strength Steels, October 20, 1961
133	Titanium in Aerospace Applications, October 24, 1961
134	Machining of Superalloys and Refractory Metals, October 27, 1961
135	Review of Recent Developments in the Technology of Nickel-Base and Cobalt-Base Alloys, October 31, 1961